# EXTRACTION OF GAHARU ESSENTIAL OIL USING ULTRASONIC ASSISTED STEAM DISTILLATION

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**A thesis submitted in fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering**

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I declare that this thesis entitled "*Extraction of Gaharu Essential Oil Using Ultrasonic Assisted Steam Distillation*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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*Special dedication to my family members that always inspire, love and stand besides me, my supervisor, my beloved friends especially the one who always help me, my fellow colleagues,*
*and all faculty members*

*For all your love, care, support, and believe in me. Thank you so much.*

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## ABSTRACT

*Aquilaria* species from the family of Thymelaeceae are the main source of gaharu, which has been classified as one of the most highly valuable, non-timber products in the world market. Its distinctive fragrance has been valued in many cultures and it is widely used in religious ceremonies, medication, incense, perfume and toiletry products. Currently, the method used for extracting gaharu essential oil is by using hydrodistillation. However, this method is inefficient which it produced low yield of oil and longer time of extraction and thus increasing the production cost. To overcome those problems, this study will be conducted to improve existing method of extraction by using ultrasonic assisted steam distillation. Parameters involve in this study is pretreatment time and gaharu-to-water ratio and both are manipulated to gain high yield of oil with optimum and maximum. The results from this study is the gaharu essential oil yield is increasing with the increment of both pretreatment time and gaharu-to water ratio until it reached a condition where the yield of oil become constant. The best pretreatment time obtained is at 9 hours with oil yield of 0.1276% and the gaharu to water ratio of 1:20.which gave 0.1295% oil yield. It is approved that ultrasonic assisted steam distillation is feasible to improve current method of gaharu essential oil extraction by gaining high oil yield and saving production cost.

## ABSTRAK

Spesis *Aquilaria* dari keluarga Thymelaeceae adalah sumber utama penghasilan gaharu yang telah diklasifikasikan sebagai hasil bukan kayu paling berharga di pasaran dunia. Harumannya yang unik telah dihargai oleh banyak kebudayaan and ia telah digunakan secara meluas di dalam upacara keagamaan, perubatan, setanggi, wangian dan barangan pembersih. Pada masa ini, kaedah yang digunakan untuk pengekstrakan minyak pati gaharu adalah dengan menggunakan penyulingan air. Tetapi, kaedah ini tidak efisien kerana ia menghasilkan hasil minyak yang sedikit serta masa pengekstrakan yang panjang sekaligus meningkatkan kos penghasilan. Untuk mengatasi masalah tersebut, kajian ini akan dijalankan untuk meningkatkan kaedah asal pengekstrakan dengan menggunakan penyulingan wap dibantu *"ultrasonic"*. Parameter yang terlibat dalam kajian ini adalah masa pra-rawatan dan kadar kayu gaharu per isipadu air dan kedua-duanya dimanipulasi untuk mendapat hasil minyak pati yang banyak dengan pengekstrakan minyak yang optimum.dan maksimum. Keputusan yang dijangkakan dari kajian ini adalah hasil minyak pati gaharu meningkat dengan peningkatan masa pra-rawatan serta kadar kadar kayu gaharu per isipadu air sehingga ia mencapai keadaan di mana hasil minyak menjadi tetap. Masa prarawatan yang terbaik diperoleh pada 9 jam dengan menghasilkan 0.1276% pengeluaran minyak dan kadar kayu gaharu per isipadu air terbaik adalah pada nisbah 1:20 yang menghasilkan 0.1295% pengeluaran minyak. Adalah diharapkan bahawa penyulingan wap dibantu "*ultrasonic*" akan memperbaik kaedah pada masa ini untuk mengekstrak minyak pati dengan memperoleh hasil minyak yang tinggi dan menjimatkan kos penghasilan.

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Research

Gaharu is the resinous, fragrant and highly valuable heartwood produced by *Aquilaria malaccensis* and other species of the Indomalesian tree genus *Aquilaria*, from the family of Thymelaeceae. There are fifteen species in the *Aquilaria g*enus and eight are known to produce gaharu. In Malaysia, gaharu is primarily produced from *A.malaccensis, A.hirta, A.microcarpa, A.rostrata* and *A.beccariana* (Chang *et al.*, 2002) and they are large evergreen trees growing over 15-30 m tall and 1.5-2.5 m in diameter, and has white flowers (Chakrabarty *et al.*, 1994). The taxonomy of gaharu tree is shown below in Table 1.1.

**Table 1.1 :** The taxonomy of gaharu tree

| | |
|---|---|
| KINGDOM | Plantae |
| PHYLUM | Tracheophyta |
| CLASS | Magnoliopsida |
| ORDER | Myrtales |
| FAMILY | Thymelaeaceae |
| GENUS | *Aquilaria* |
| SPECIES | *malaccensis* |
| SPECIES AUTHORITY | Lamk. |

(Source: International Union for Conservation of Nature and Natural Resources)

Gaharu is known under many names in different cultures: Gaharu in Malaysia and Indonesia, Chen-xiang in Chinese and Jin-koh in Japanese that both literally mean “sinking incense” due to its high density of oleoresin, Oudh in Middle East, Agarwood, Aloeswood and Eaglewood.

**Figure 1.1** *Aquilaria malaccensis* Plantation in Thailand
(Source: Robert A. Blanchette)

The formation of gaharu is a natural process in response to a parasitic ascomycetous mould, *Phialophora parasitica*, a dematiaceous (dark-walled) fungus attacks (Gibson, 1977 cited in Ng *et al.*, 1997) and the aromatic resin is usually formed in the bark and the roots as well as the heartwood of the trees (Jalaluddin, 1977 cited in Donovan and Puri, 2004). As a response, the tree produces a resin high in volatile organic compounds that aids in suppressing or retarding fungal growth. While the unaffected wood of the tree is relatively light in colour, the resin dramatically increases the mass and density of the affected wood, changing its colour from pale beige to dark brown or black (Rahman and Basak, 1980 cited in Blanchette and van Beek, 2005). The uninfected gaharu has no scented value. In natural forest only 10% of the trees are infected by the fungus. A common method in artificial forestry is to inoculate, a forced method where the trees are deliberately wounded, leaving them more susceptible to a fungal attack.

Gaharu has been known to possess medicinal properties as well. Its use as a medicinal product has been recorded in the *Al-Hadith Al-Sahih Muslim,* which dates back to approximately the eighth century, and in the Ayurvedic medicinal text the *Susruta Samhita*. It is used in Ayurvedic, Tibetan and traditional East Asian medical practices (Barden *et al.,* 2000) as an alternative to modern pharmaceutical products.

The high value of gaharu makes it become the good choice for collection and investment. The larger piece of some gaharu which possesses special artistic shape or natural carving is precious and its value is exceptional.

The odour of gaharu is complex and pleasing, with few or no similar analogues. Due to its aromatic properties, gaharu has been widely used for thousand of years in Middle East, China, Japan, India and Indochina, notably in religious purposes by Buddhists, Hindus and Muslims either in the form of essential oil or incense sticks.

Its distinctive fragrance has been valued in many cultures, thus it is used by direct burning the chips or pieces of gaharu, slow-burning incense sticks and perfumes. Moreover, its use as a perfume has been recorded in the *Old Testament.* In East Asia, Indochina and Tibet, gaharu is used extensively by the monks as an aphrodisiac and calming of minds.

Majority of genuine essential oils are extracted by distillation method. The production of gaharu essential oil has become the cottage industry in East of Peninsular Malaysia and also traditionally distilled by the indigenous people mainly by using water distillation or hydrodistillation. It is the oldest form of essential oil extraction and is believed by many to be the only way oils should be extracted.

Nowadays, the research on gaharu and the extraction of essential oil have been done actively by research institutes and local universities in order to find the best method that yield the maximum essential oils with best quality and high value with energy and costs savings. In Malaysia, the scientific works have been done by

Forest Research Institute of Malaysia (FRIM) and several public higher learning institutions.

## 1.2 Problem Statement

Currently, the method of extracting gaharu essential oil is using traditional water distillation method (Chang *et al.*, 2002) or hydro distillation. This method involves submerging the desired raw material (gaharu chips or powders) in water in the still and brought to boil, the oil that evaporates is lost in the water in the still as well as in the aqueous phase of the distillate. The residual oil dissolved in the water usually causes odour nuisance when it degrades and is also a waste of the valuable product in the water stream (Masango, 2005).

Besides, this extraction method acquires long extraction times that consume a lot of fuel for heating purposes. The extraction process did not produce the maximum yield of oil because the efficiency of the method itself is relatively low. All this will result in higher operating cost especially for heating process (A. Fadzli, 2006).

Extraction process using ultrasonic assisted steam distillation method may contribute to improve the efficiency and capacity of gaharu essential oil extraction. This approach will be applied in this research to examine the feasibility of ultrasonic assisted steam distillation as an improved method for gaharu essential oil extraction process.

## 1.3 Objective of Research

To examine the feasibility of ultrasonic assisted steam distillation as an improved method for gaharu essential oil extraction process.

## 1.4 Scopes of Research

In order to achieve the objective, these following scopes have been identified and to be applied:

i. To study the effect of ultrasonic pre-treatment time on the gaharu essential oil yield.
ii. The study the effect of solid to solvent ratio on the gaharu essential oil yield.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Gaharu

### 2.1.1 Overview of Gaharu

*Aquilaria spp*. (Thymelaeaceae) are the principal source of Gaharu (Soehartono and Newton, 2001), a resin-impregnated heartwood that is fragrant and highly valuable. Other names used by both collectors and traders of the fragrant wood are agar, aloeswood, eaglewood, kalambak or gaharu depending on the country and generally encompass the fragrant wood produced by most species of *Aquilaria* (Ng *et al.*, 1997).

In Malaysia, the tree of *Aquilaria* is called karas and its fragrant wood is known as gaharu. Five species of *Aquilaria* are recorded for Peninsular Malaysia and all are believed to be able to produce oleoresins. The most popular species generally associated with gaharu is *A.malaccensis* (Chang *et al.*, 2002).

*A.malaccensis* is widely distributed in south and south-east Asia. According to Oldfield *et al.* (1998), *A.malaccensis* is found in 10 countries: Bangladesh, Bhutan, India, Indonesia, Iran, Malaysia, Myanmar, Philippines, Singapore and Thailand. *Aquilaria* species have adapted to live in various habitats, including those that are rocky, sandy or calcareous, well-drained slopes and ridges and land near

swamps. They typically grow between altitudes of 0-850 m, in locations with average daily temperatures of 20-22 °C (Wiriadinata, 1995).

*A.malaccensis* is distributed throughout Peninsular Malaysia, except in the states of Perlis and Kedah (Whitmore, 1990), and is known to produce medium-quality grade gaharu (Burkill, 1966). Gaharu is frequently found as irregular patches or streaks in the wood of about 20 years old trees. Very often, the quantity and quality of gaharu produced increase with age, with the best yields occurring in trees aged 50 and above (Sadgopal, 1960).

### 2.1.2 Formation of Gaharu Resin

Research conducted so far has focused mainly on the following three hypotheses exist regarding gaharu formation, namely that it is the result of diseased or pathological, wounding/ pathological and/ or non-pathological processes (Ng *et al.*, 1997). The pathological condition was first hypothesized by fungal infections that lead to diseased wood. Moreover, the wounding/ pathological condition considered that wounding has a primary effect on gaharu formation with fungal infection as a secondary influence (Gibson, 1977; Rahman and Basak, 1980). Ng *et al.* (1997) suggested the non-pathological condition is a defensive response of the tree towards wounding, therefore release the gaharu resin.

The infection of fungi occurs when branch and stem injured by larvae of mainly a parasitic ascomycetous mould, *Phialophora parasitica*, a dematiaceous (dark-walled) fungus (Gibson, 1977 cited in Ng *et al.*, 1997). It is seen that the larvae of *P. parasitica* bore the standing tree trunk of *A.malaccensis* and make tunnels inside the tree trunks. Fungus enters the plant through this vertical hollow sometimes-zigzag tunnel inside the stem, which serves the initial sites of infections. Later on infections spread an all sides slowly and gradually and ultimately a larger wood volume gets infected. More insect infestation in the infected area more is the chances to form gaharu in 7-8 years time after infection. Gaharu formation is the resinification of accumulated oleoresin due to the action of microorganisms. Figure 2.1 shows the scanning electron microscopy of a cross section of decayed tree which

contain abundant amounts of resin formed in the wood cells. *Aquilaria* has an unusual anatomy and specialized cells within the xylem produce the gaharu resin.

Infections may also occur due to mechanical or natural injuries on the stem, for example following wind or storm damage. Due to infections, oleoresins are accumulated in the infected wood and later become odoriferous. At the initial stage infections appear as brown streaks in the tissue. Accumulation of oleoresins goes on increasing with the increase of infection rate as well as aging of the infection. As more of oleoresins are deposited the intensity of colour of the infected wood increases and finally it becomes black due to increase in concentration. Figure 2.2 shows the cross section cut of the tree showing dark regions of gaharu formed in the heartwood.

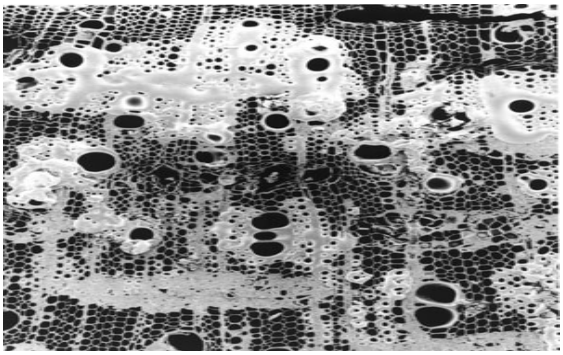

**Figure 2.1** Abundant amounts of resin formed in the wood cells
(Source: Robert A. Blanchette)

The fungal infection takes long time to mature and trees about 50 years old have the highest concentration between 2.5-5.0 kilogram/ tree. Furthermore, other factors such as environmental variation, within-tree seasonal variation in responsiveness and the possible existence of two or more varieties or genetic strains in *Aquilaria spp*. may also play an important role in the formation of gaharu (Ng ***et al.***, 1997).

**Figure 2.2** Cross section cut shows gaharu formed in the heartwood
(Source: Robert A. Blanchette)

However, the Tropical Rainforest Project (TRP) in Vietnam has found that *Aquilaria malaccensis* can be artificially induced to yield gaharu at a ten times faster than in natural formation (Barden *et al.*, 2003). Figure 2.3 shows the artificial wounding made to the trunk of *Aquilaria* tree in order to generate more gaharu resin production.

**Figure 2.3** Artificial wounding to generate the production of gaharu resin
(Source: Yip and Lai, 2005)

### 2.1.3 Harvesting of Gaharu

Since gaharu is located in the heartwood of the *Aquilaria spp.*, its detection from outer appearance is not easy. Generally, such trees are distinguished by certain

external features whether or not the tree harbours precious gaharu oil or gaharu deposits. These include:

i. a poor crown, decayed branches, and uneven bole;
ii. swelling or depressions and cankers on the bole;
iii. the appearance of hordes of ants in the fissures;
iv. a distinctly yellowish to brownish trace in the wood under the outer bark; and
v. signs of ill-health particularly a die-back symptom of the top and outer branches and a yellow tint to the woody tissues.

The visible wounds, cankers on the bole, stem distortions, smaller leaves and the rotten branches provide evidences of gaharu deposits within a tree. Wood assumes distinctly yellowish trace when gaharu formation takes place. The normal wood in the healthy trees is of pale brown-beige colour. The change can be observed by removing the bark of the tree. Sometimes screw augers are driven inside at various depth and samples are drawn for examination. Finally the odour on the examination by drawing samples with the help of screw augers. The disease or the fungal infection usually takes some time to make it manifest, hence gaharu is hardly found in young shrubs (Anonymus, Hand Book On Medicinal & Aromatic Plants).

In Peninsular Malaysia, Orang Asli is the traditional harvester of gaharu. Over in East Malaysia, Penans are the traditional gatherers of gaharu. Gaharu harvesting is a destructive process; however they will not fell a tree unless he is certain of the presence of good quality gaharu in the tree. Slash marks on standing karas trees made by the harvesters to check for the presence of gaharu (Hansen, 2000 cited in Chang *et al.*, 2002).

Dayak communities in Indonesia believe that dying seedlings and saplings (indicated by yellowish leaves) testify to infection of the mother tree. They appear to be able to identify infected trees by differentiating between the sound made by knocking on the infected trunks and the sound made by knocking on non-infected trunks (Soehartono and Mardiastuti, 1997).

### 2.1.4 Grading and Pricing of Gaharu

There are five major criteria to grade the gaharu which are the buoyancy, fragrance and medicinal property, colour and reasons of forming (http://www.jdcorp.com). The detailed descriptions as shown as below:

1. Buoyancy

The most important and objective criterion to grade the genuine *Aquilaria* is to measure its buoyancy. *Materia Medica* and *Description of the Plants in Southern China* stated that there are three grades for *A.agallocha* according to its buoyancy whether they are complete sinking, half sinking (floating under the surface of water), and floating kinds.

The density of *Aquilaria* tree without oleoresin is a mere 0.4. However, when the percentage of containing oleoresin is over 25%, gaharu in any forms (chips, powders, or larger pieces) will sink into water. The Chinese name of gaharu is originated from this characteristic.

In Japan and Korea, only *Aquilaria* that contains more than 25% of the oleoresin can be used as medicine. This regulation is based on the fact that the only sinking *Aquilaria* can be used as medicine. In China, 15% is the minimum requirement.

2. Fragrance and Medicinal Potential

The characteristics of Aquilaria are acrid, bitter and warm. These characteristics can also apply its medicinal potency and fragrance. One important characteristic of gaharu is that it does not have noticeable fragrance before burning.

Different from most raw materials, gaharu possesses strong ‘natural’ antibiotic function. The higher the grade of gaharu, the more effective and ‘warmer’ the curing process, and the warmer and the richer flavor it has.

Generally, gaharu with different colors has different fragrance. However, grading gaharu by its fragrance is quite difficult as individual preferences of fragrance differ. Therefore, judging grade simply by its fragrance inevitably will result in subjective judgment.

3. Colour

Gaharu oleoresin presents in several different colors. Examining the color of gaharu should be done under natural sun light. There were various researches about the color of gaharu.

There are five colour grading for gaharu. The highest grade is Green; Dark Green comes second, and then Golden (light yellow), Yellow and Black.

It's generally believed that the color gaharu oleoresin is black. In reality, gaharu containing higher percentage of oleoresin usually shows green or dark green luster.

4. Gaharu’s reason of formation

There are four reasons of gaharu oleoresin's formation shows in most studies. Different formation results in differences on colors, fragrances, and containing of gaharu resin.

The best gaharu is formed naturally inside the live *Aquilaria* without any wounds. The second grade gaharu is formed after the tree is died. The third